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A REVIEW OF THE SOL-GEL PROCESS AND ITS APPLICATION

Subhayu Choudhury

Assistant Professor, Department of Physics, Santipur College, Nadia, West Bengal

ABSTRACT

The sol-gel process has been very useful to produce nanomaterials using different precursors since the early days. Precursors like metal alkoxide or organic/inorganic hybrids are dissolved in alcohol or water and converted into gel after alcoholysis or hydrolysis. The low temperature and low cost of the sol-gel technique make it more convenient than other methods. In comparison to other method, it has good control over the homogeneity of the particles. This process is very much used in ceramics, composites, thin film coating, and other industrial purposes. This review is a study about the process and application of sol-gel-derived materials. The historical point of view of this process is also explained in this review.

KEYWORDS: Nanomaterials, Sol-Gel, Alcoholysis, Application.

INTRODUCTION

Nowadays, different techniques such as the vapor phase compression method, plasma method, electrochemical method, mechanical alloying method and sol-gel method are used to produce and synthesis of nanomaterial particles. Among the above methods, sol-gel methods are mostly used for industrial applications [1-4]. Using this method alloys are produced very easily because different material nanoparticles are synthesised simultaneously. Mixing two or more metal nanoparticles alloy is made in one step. Although electrochemical and plasma methods can produce alloys, the sol-gel method can produce industrial-scale nanomaterials [5]. Production cost is cheaper than other conventional methods. The sol-gel method is capable to produce highly pure and uniform composites and nanomaterials. This method is capable to produce nanomaterial in lower temperature (700^0 C-3000^0 C) compared to other method which is very much advantageous [6]. This method is a purely chemical process through different irreversible processes a product is produced [7].

The sol-gel method is a bottom-up approach method. The first process is to produce a colloidal solution of precursors. After that, it transforms into a gel, made of discrete particles of polymers. Precursors are hydrolysed and poly-condensed to produce colloids. A schematic diagram is shown in Figure 1[8]. Sol-gel-derived products are very much used in thin film technology to make film. This thin filmmaking is very much easier and cheaper compared to others. Sol-gel-derived materials are very much applied in various fields such as space, energy, sensors, electronics, medicines mainly drug therapy glass industry etc. In this review, the mechanism of sol-gel will be discussed and also the application of sol-gel-derived materials in different fields will be highlighted.

Sol-gel: Colloidal solution is suspended small-sized particles in a liquid. After that, 3d complex gel network has formed i.e, the gel is formed.

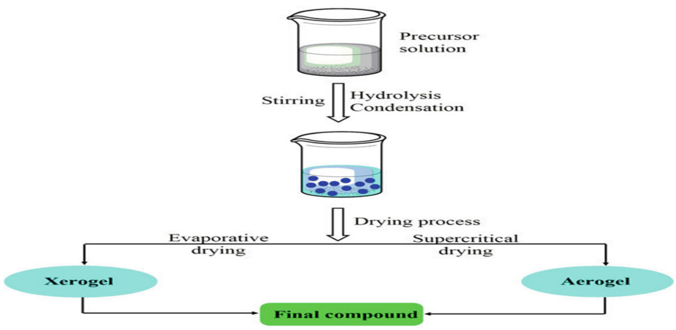

Figure 1: Schematic diagram of sol-gel process[8]

The sol-gel process can be divided into various steps-

Mixing: Precursors are mixed mechanically. One of the precursor metals alkoxide always reacts with water. Then it is hydrolysed and polycondensation and produces a colloidal solution i.e. sol.

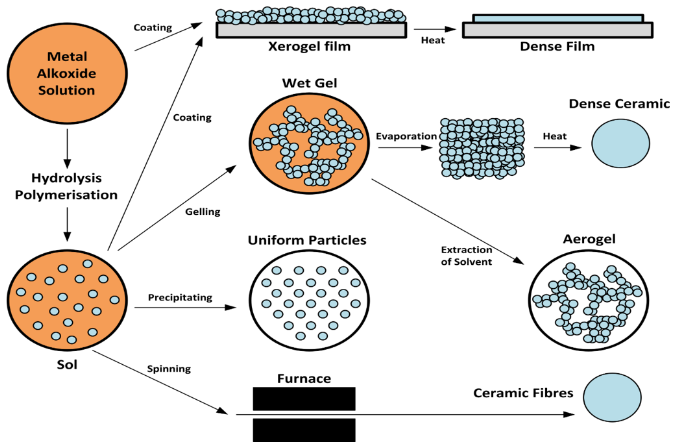

Figure 2: Scheme of sol-gel technique [9]

Gelation formation:
After the solution three-dimensional network has formed. This gelation process takes place through hydrolysis and polycondensations[10]: -

Hydrolysis:
Hydrolysis happens for metal alkoxide precursors. In this process OR^- group will be replaced by OH^- ion of water. As the reaction is slow for the neutral case so bare or acid catalysts needed to speed up the reaction. In both cases are shown in Fig 2. In both cases reaction is faster than the acidic case.

$$M(OR)_4 + H_2O = M(OH)_4 + 4R(OH)$$

Polycondensation:
Condensation may happen during hydrolysis or after completion of hydrolysis.In condensation chain like bond is formed and produce a colloidal soln.

$$M(OH)_4 + M(OH)_4 = 2MO_2 + 4H_2O$$
$$M(OH)_4 + M(OR)_4 = 2MO_2 + 4ROH$$

In acidic conditions, condensation begins before hydrolysis is complete. After sol, it forms a network-like gel which is shown in Fig 3. The gelation process depends upon other factors such as water and metal reaction.

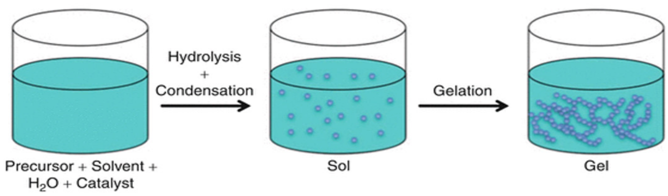

Figure 3: Scheme of gelation [11]

Aging: In this step gel is rested for hours to days. During this process porosity of the gel will be decreased and increased thickness of the interparticle. The final product of aging is resistive to cracking while drying.

Drying:
This step is very important in this process. Excess solvent is evaporated in this step. During this step, capillary stresses can develop around pores. This stress causes the cracking of gel. Gels become low-density after drying. It is very thermal insulation when it is placed between the glass plates.

Densification:
After drying gel is heated in a furnace at elevated temperature. This heat makes the gel densified. The elevated temperature depends on pore dimension, pore correctivitty and its surface area.

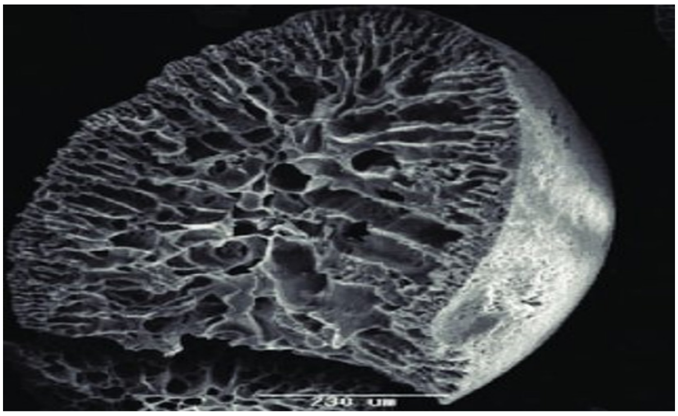

Figure 4: SEM image of a dried gel after the sol-gel process: this image confirms the presence of nanometer porosity with a very high specific surface area[12]

Historical point of view:
Sol-gel technology was used in the old days[13]. But Ebelman first showed the formation of transparent material by slow hydrolysis of an ester of silica acid in 1845[14]. Faraday in 1853 produced a solution from colloidal gold particles in a lab. Sol-gel is not an artificial thing it, blood is a natural example of sol-gel. The development of sol-gel science has increased rigorously as it produced nanoparticles and nanocomposites [15-17]. Sakka is the main pioneer in this field[18-20]. Among all other composite silicates are researched mostly. Lamar explained beautifully the formation of colloidal particles by proper growth from appropriate precursors. After that Majaric applied this information to grow homogenously sized particles coagulation or stabilization of sol is mainly controlled by electrostatic interaction [21].Derjaguin,Verway,landau and Overbeck gave theory about the aspects of colloids, which brought them Nobel prizes in Physics[22]. Flory also won the Nobel prize in Chemistry due to his historic theory on the Behaviour of polymer solution. Stockmayer and Flory gave the function of the gelator phenomenon about polymer solution undergoing growth by condensation reaction[22]. Gel is defined as the formation 3D continuous network depending upon the size of the container. However, the critical value of gel does not rely on the size of the container. Mandelbrot showed the gel complex architecture after gelation in fractal geometry. Diffusion limited aggregator was also proposed for dense colloidal particles .

Advantages of the sol-gel technique:
In sol-gel derived materials are highly pure materials. It is low cost technique compare to other technique and it is processed at low temperature. Alloys can be formed easily using different processes. The coating is easier for so-gel-derived sol. This method has several advantages in forming metal oxide nanoparticles compared to others. Highly pure powder can be made with homogeneous molecular mixing of the raw components. Some of the major advantages [23-25] are —

- Creating metal oxide nanoparticles.
- Low cost and highly chemical product composition.
- Processing of highly pure products.
- Making of the layer of amorphous materials.
- Highly surface coverage.

- Geometrically shaped optical components.
- This method can be used to make ceramics and as a transition layer between thin films of metal oxides in a variety of applications.

Disadvantages Of sol-gel: It cannot be used for industrial purposes. It takes a longer time to make the final product and metal alkoxide precursors are costly [26-30]. Another drawback of this process is the understanding of the reaction [31].

Parameters of sol-gel method:
Sol-gel method depends upon different factors as PH, solvent, Temperature, agitation, Time and catalysts which are discussed below---------

- PH:- Sol-gel chemistry very much depends upon the PH of the solution [32]. XRD pattern and SEM images of ZnO particles for different PH levels are shown in fig 5 and figure 6. respectively.

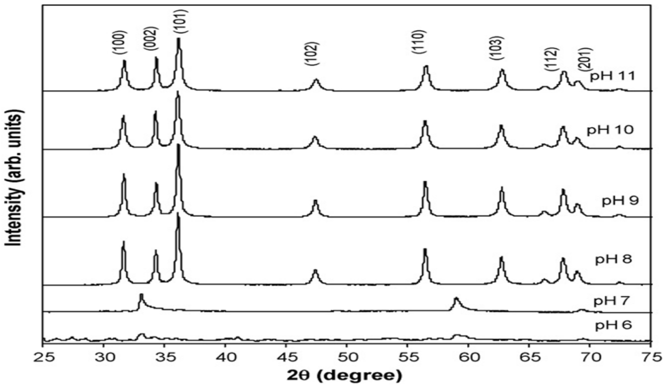

Figure 5: XRD pattern of ZnO particles for different PH [32].

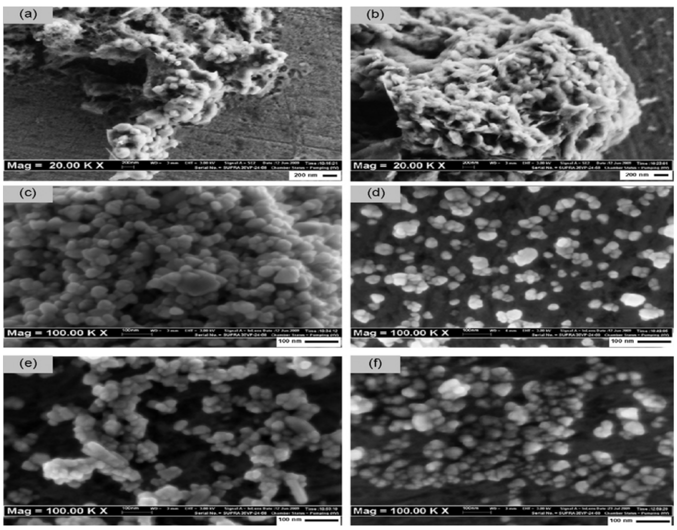

Figure 6: FESEM images of ZnO powders synthesized at different PH values a)PH-6 b)PH-7 c)PH-8 d)PH-9 e)PH-10 f) PH-11[32].

- Solvent: Solvents are very important during the polycondensation process. First, it helps NPs to keep dissolved.
- Agitation: During gel formation gel networks can be formed domain-wise. Mixing will break the domain-wise networks and thereafter it will form a leigh network. Agitation also helps the chemical reaction in all parts of the solution and allowing all molecules to receive adequate amount of chemicals.
- Time: Time takes part in very much in different gelation processes because the solution takes different times to form gels. Gelation is a slow process. Accelerating reaction for a short time sometimes cannot form a gel.
- Temperature: The gelation is a slow process. Temperature accelerates the chemical kinetics of the reaction in the formation of gel. Sometimes gelation needs time for a week or month. Adequate temperature reduces the time in the formation gel. It also helps to bind NPs to form a gel.
- Catalysts: Catalysts play a big role in chemical reactions to speed up. Because acidic (H+) and base are catalysts in this case work differently to accelerate the reaction and reduced time to form gel.

Application:
Sol-gel technique is very often used to produce metal oxide nanoparticles like TiO_2, ZnO_2, WO_3, and SnO_2 which have different applications in many fields shown in fig [33].The special properties of composites made of TiO_2 and V_2O_5 have led to their usage as supercapacitor electrodes [34, 35]. It is reported that TiO_2 nanoparticles can be applied for treating cancer[36].

Sol-gel method is used to create ceramics easily. Ceramics materials are non-metallic, low density and ductile. The final product will be corrosion resistive, hardness and wear resistance. Most ceramics are aluminium and silicon carbide.

Sol-gel-derived films have many advantages over conventional cases. Ceramics are less dense than metallic alloys. Ceramics are brittle and lack toughness.

Sol-gel-derived products is very much useful for producing thin films of TiO_2, SiO_2, and MgF_2. This film can be applied on metal, crystal, glass or ceramics to change optical property. This coating will help reflection, refraction, conductivity and antireflection. Many important devices depend upon thin film coating such as solar panels, glass, flat tv panels, spectacle lenses, smartphones etc. Plastic substrates are often used for coating thin film. Then it becomes cost-effective and light-weight [37].

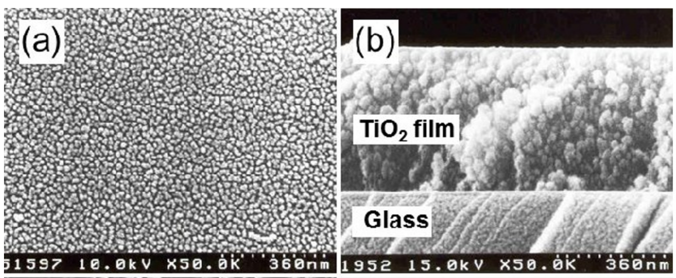

Figure 7: FESEM images of TiO2 thin film a) top view b) cross-sectional view [38].

As the sol-gel method can produce nano-size materials which

has a high surface-to-volume ratio. These Q-dot films have unique properties from their bulk nature. So, it is used in memory devices, optoelectronic devices and bio-sensors.

Porous Films: The sol-gel method can produce porous films which has large surface areas. So, this is very much used in solar cells such as TiO_2 thin film have large surface area.

CONCLUSION:

Sol-gel process is a low-cost process but it is a slow procedure. It is a low-temperature process in nature. Through this technique, we can achieve nano order particles with pure and better homogeneity than other process. It is very much suitable for thin film-making process other than the sputtering process. Besides film coating, it is used for shape casting and fibre pulling.

REFERENCES

1. A. Feinle, M. S. Elsaesser, and N. H¨using, "Sol-gel synthesis of monolithic materials with hierarchical porosity," Chemical Society Reviews, vol. 45, no. 12, pp. 3377–3399, 2016.
2. M. Niederberger and N. Pinna, Metal Oxide Nanoparticles in Organic Solvents: Synthesis, Formation, Assembly and Application (Engineering Materials and Processes), Springer, Berlin, Germnay, 2009.
3. Y. Liao, Y. Xu, and Y. Chan, "Semiconductor nanocrystals in sol-gel derived matrices," Physical Chemistry Chemical Physics, vol. 15, no. 33, Article ID 13704, 2013.
4. G. J. Owens, R. K. Singh, F. Foroutan et al., "Sol-gel based materials for biomedical applications," Progress in Materials Science, vol. 77, pp. 1–79, 2016.
5. M. Haruta, "Nanoparticulate gold catalysts for low-temperature CO oxidation," Journal of New Materials for Electrochemical Systems, vol. 7, pp. 163–172, 2004.
6. B.G. Rao, D. Mukherjee, B.M. Reddy, Chapter 1—novel approaches for preparation of nanoparticles, in Nanostructures for novel therapy, ed. by D. Ficai, A.M. Grumezescu (Elsevier, Amsterdam, 2017), pp. 1–36
7. S.M. Gupta, M. Tripathi, Cent. Eur. J. Chem. 10, 279–294 (2012)
8. Z.Nadia, Nanocomposite Materials Based on TiO2/Clay for Wastewater Treatment: A Study in Large Companies,2019 p.363-380
9. http://www.spectraresearch.com/news.html, (2015)
10. A. E. Danks, S. R. Hallb and Z. Schnepp, The evolution of 'sol–gel' chemistry as a technique for materials synthesis, Mater. Horiz., 2016
11. C.J. Brinker, G.W. Scherer, SOL-GEL SCIENCE: The Physics and Chemistry of Sol-Gel Processing, ACADEMIC PRESS, INC.
12. D. Bokov, A. Turki Jalil, Nanomaterial by Sol-Gel Method: Synthesis and Application, Advances in Materials Science and Engineering,2021
13. Y. Dimitriev, Y. Ivanova, R. Iordanova*,HISTORY OF SOL-GEL SCIENCE AND TECHNOLOGY (REVIEW), Ivanova, R. Iordanova 181 Journal of the University of Chemical Technology and Metallurgy, 43, 2, 2008, 181-192
14. J.J. Ebelmen, Untersuchungen über die Verbindungen der Borsäure und Kieselsäure mit Aether, Justus Liebigs Annalen der Chemie, 1846, 57(3), 319-355
15. B.C.Dave,S.B.Blakwood, Sol-Gel Method,Encyclopedia of nanotechnology,2016,p.3748-3761
16. Proceedings of XVII international congress on Glass,Glass surface,Sol-gel glasses ed Fongtion,Chinese ceramic society Beijing 1995
17. Proceeeding of the XX international congress on Glass,Ceramic society of Japan,Kyoto,2004.
18. S.Sakka,Trans,Indian ceramic society,46,1,1987,1
19. S.Sakka Am.ceramic Bull64,1985,1463
20. S.sakka,J Sol-gel Sci and Tech..37,2006,135.
21. Matijevic E., Butterworth Heinemann, London, 39-59 (1994)
22. A.C Pierre, Sol Gel Technology, (2000).
23. Amit Kumar, Nishtha Yadav , Monica Bhatt, Neeraj K Mishra, Pratibha Chaudhary and Rajeev Singh, Sol-Gel Derived Nanomaterials and It's Applications: A Review, Research Journal of Chemical Sciences ISSN 2231-606X Vol. 5(12), 98-105, December (2015)
24. Pratikhya Badanayak and Jyoti V Vastrad, Sol-gel process for synthesis of nanoparticles and applications thereof, The Pharma Innovation Journal 2021; SP-10(8): 1023-1027
25. A. Feinle,a M.S. Elsaessera and N. Hüsinga, Sol-gel synthesis of monolithic materials with hierarchical porosity,The Royal Society of Chemistry 2012.
26. Deng Z, Wang J, Wei J, Shen J, Zhou B and Chen L, J Sol Gel Sci Technol, 19, 677-680 (2000)
27. Venkastewara Rao A, Bhagat S.D, Hirashima H and Pajonk GM, J Colloid Interface Sci, 300, 279-285 (2006)
28. El Rassy H, Buisson P, Bouali B, Perrard A, Pierre AC, Langmuir, 19, 358-363 (2003)
29. Harrels JH, Ebina T, Tsubo N and Stucky G, J NonCryst Solids, 298, 241-251 (2002)
30. Allie C, Pirard R, Lechloux AJ, Pirard JP, J Non-Cryst Solids, 246, 216-228 (1999)
31. Mackenzie J.D., Journal of Non-Crystalline Solids, 100, 162-168 (1988)
32. S.S. Alias, A.B. Ismail, A.A. Mohamad, Effect of pH on ZnO nanoparticle properties synthesized by sol–gel centrifugation,Journal of alloys and compounds 2010.
33. Mritunjaya Parashar· Vivek Kumar Shukla · Ranbir Singh, Metal oxides nanoparticles via sol–gel method: a review on synthesis, characterization and applications. Journal of Materials Science: Materials in Electronics(2020).
34. A. Ray, S. Roy, S. Saha, Das, Transition Metal Oxide-Based Nano-materials for Energy Storage Application. Science Technology and Advanced Application of Supercapacitors (INTECHOPEN, London, 2019)
35. S. Sasirekha, G. Arumugam, Muralidharan. Appl. Surf. Sci. 449, 521–527 (2018)
36. H.M. Yadav, N.D. Thorat, M.M. Yallapu, S.A.M. Tofail, J.-S. Kim, J. Mater. Chem. B 5, 1461–1470 (2017).
37. Choi A.H. and Ben Nissan B., Journal of crystalline Australian Ceramic Society, 50, 121-136 (2014)
38. H.J.KIM,S.M.Yoo,S.Yu,W.Lee,Photocatlytic Behavior of TiO2 films:Thickness and roughness dependence,Rapid communication in Photoscience(2013)vol 2, no-1 ,pp 1-8